

Step-by Step Description of Buffered Chemical Polishing at ANL-FNAL BCP Facility

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Hydraulic scheme of the BCP facility is shown in the picture in the end of the document. This document provides information needed for development a control system that must not only follow right sequence of etching steps, but also implement certain safety features that make work of involved personnel less stressful and safer.

All the process is divided by several cycles, namely:

1. System Preparation,
2. Etching
3. Rough Rinse
4. Final Rinse

Each cycle is performed in automated mode, but transition between the cycles must be confirmed by an operator of the system. In a case if something is going wrong in the system, it provides the operator with a warning and a choice of options to follow. If no choice can be made, the operator can switch to manual mode of operation to complete the process or safely interrupt it.

The following is a systematic description of the process that is followed by a recommended way of dealing with possible equipment failures.

If no etching is scheduled in several days after the last one is complete, etching compartment and major equipment must be cleaned and dried to avoid any contaminants from accumulating. This procedure is out of the scope of this document and must addressed after the facility is assembled at ANL.

System Preparation.

The goal of this stage is to prepare the system for etching, to check all elements of the hydraulic scheme, and to be sure the control system works as is required. The testing must be performed fully after the system is modified, or repaired, or maintenance operations is performed on the system, or it was idle for a long time. It is possible to skip some steps if the process is made on a regular basis, and the system condition and behavior is well known. The steps during the system testing stage must reproduce BCP process with water substituting for acid.

1. Pre-requisitions:

- UPW storage tank T10 (located outside of the process room is filled with UPW, which is circulated through the system on the permanent basis. Water level in T10 is controlled automatically, and UPW system has the capacity to fill the tank between the rinsing cycles.
- the hydraulic system must be cleaned and rinsed using UPW before the testing starts;
- tanks T1 – T5 must be empty before the process starts;
- all the valves are closed initially.

2. Fill the tank T2 with needed amount of UPW by opening V12. Check readings of L2 and R2.
3. Bring cavity-jacket assembly in and position it the etching tank T5 (see specification # XXXXXX). Before cavity is installed in the etching tank, the jacket (tank T6 in the scheme fig. 1) must be filled with UPW and checked for leaks. Make needed hydraulic connections in accordance with the hydraulic scheme MD-XXXXXX.
4. Bring in the process room a barrel filled with UPW and make needed connections for acid cooling. **Block V14, V15, V17 and V18 in the closed position !!** Open V13, and V19. Start Pmp1. Check flow meter FM1 and readings of the pressure gauges Pr1 and Pr2.
5. Start circulating water through the jacket by starting the pumps Pmp6. Check work of flow meter FM6. Check R6 and TG6 readings.
6. Start the water chiller WC. Circulation pumps Pmp7 must start simultaneously. Check WC coolant temperature setting (TG8). Check temperature response of the gauges in the HE tank T7 (TG7) and cavity jacket T6 (TG6) circuits. Check readings of the flow meter FM8.
7. Fill the tank T1 with an appropriate amount of UPW from the barrel: open V16, close V13, and watch the readings of the level meter L1. Make sure L1 reading is consistent with the integrated reading of the flow meter FM1. **Check for leaks.** Check work of the temperature gauge TG1.
8. Add 2 gallons of UPW in the acid spill tray of T1 and check functioning of the conductivity meter R1.
9. By opening first V02 and then one by one V04 and V05, check for leaks in the acid and water piping. Open V01 (manually) to fill the process tank T5 (and the cavity) with UPW. Check for leaks. V02 must close automatically after liquid level reading in T1 (L1) reaches zero. Check functioning of the level meters L51 and L52. Open V23 and be sure V21 and V22 are closed. Start circulating water by starting the pump Pmp5. Check reading of the flow meter FM5.
10. By opening V07, dump water from the etching tank T5 into the used acid tank T3 and check it for leaks. Pmp5 stops automatically when level in T5 reaches a preset value (L52). Simultaneously V35 and V37 open to drain "acid" from the pipe. V04 and V07 are closed automatically after liquid level reading in T5 (L51) reaches zero. Check the work of the level meter L3.
11. Process tank T5 must be filled with UPW from T2 automatically by opening V03. Pump Pmp5 must starts automatically after water level in T5 reaches a preset value.
12. The valve V03 closes automatically after the gravity tank T2 is emptied. T2 must be filled with the needed amount of UPW automatically. V12 must close automatically after T2 is full (level meter L2).
13. With closed V28 and V29, open V30 and V27 and check the work of the rinsing loop T3- Pmp3-V30-V27-T3.

14. Open V28, close V27 and pump water (pump Pmp3 is on) out of T3 into the neutralization system. When the T3 is empty (L3), Pmp3 must stop automatically following by closing V28.
15. Add ~ 2 gallons of water in the room's sump pit and check work of the pump Pmp0.
16. Disconnect and remove the barrel with UPW.
17. In the end of the system preparation cycle, the tanks T1, T3, T4 are empty, tanks T2, T5 (process tank), and T6 (jacket) are filled with UPW. There is some water in the spill tray of T1. Water Chiller is on. Pumps Pmp5 and Pmp6 are on.

Etching

In the beginning of the etching cycle, the tanks T1, T3, T4 are empty, and tanks T2, T5, T6 are filled with UPW. Water Chiller is on. Pumps Pmp5 and Pmp6 are on. Be sure that UPW is added into the spill tray of the tank T1.

1. Install local hoods at the designated places in the process room. Start scrubbing system in the “**ventilation**” mode. Bring and connect to the hydraulic system a barrel with BCP mix. Bring and connect to the hydraulic system an empty barrel to contain used acid after the processing. Open V14, V15, V17, and V18, close V13, V16, and V19. **Leave the room and close the doors.** Change the mode of the scrubbing system to “**scrubbing**” and start Pmp1 to circulate acid through the heat exchanger HE. Monitor readings of FM1, FM5, FM6, FM8, TG5, TG6, TG7, TG8, R6, R7, Pr1, and Pr2.

Change in resistance reading by R7 is an indication of possible acid leak into the heat exchanger tank. Change in resistance reading by R6 is an indication of possible acid leak into the jacket. If the rate of the leak (conductivity rise) is high in comparison with the time left for the cooling stage, stop Pmp1, close all valves in the circuit, disconnect and remove acid barrels before starting a repair work. **The leak must be considered dangerous if concentration of acid in the HE tank reaches 1:250 (?). This corresponds to the initial resistivity drop rate of about ??? Ohm-cm/min.**

If readings of TG5, TG6, TG7, and TG8 do not correspond to the expected, make a decision about a possibility to proceed with the process. This choice depends on the temperature of acid in the acid barrel and on performance of the chiller. For example, if the output water temperature is close to the setting, the acid flow in the cooling circuit is probably too low. Check pressure gauges readings and switch to the bypass circuit by opening V19. If water chiller failed, stop the process and replace (or repair) the chiller. If the acid heat exchanger is leaking, replace it with a spare one (need to be in-house).

2. The cooling part of the process is over when temperature in the barrel B1 (TG5) reaches approximately (12 – 14)°C. Readings of TG6 must be at the similar level.

If the readings of TG6 are higher than 20 °C, the corresponding heat exchange circuit is not functioning properly. In this case, lower acid temperature in B1 to about 10 – 12 °C to compensate for the additional heating.

3. Open V16 and then close V14, V15 to fill the acid gravity feed tank T1. Monitor readings of L1, R1, and TG1. Compare integrated reading of FM1 with the readings of L1. Pump Pmp 1 must be controlled both by L1 and integrated FM1; it must stop when the preset acid level in T1 is reached.. Close V16.

If level monitor does not function, it is still possible to fill the tank in manual mode relying on the readings of one of them and visually using control marks on the wall of the gravity tank.

In the case when the process must be interrupted after T1 is filled fully or partially, open V13 and stop Pmp1. The acid will drain back to the barrel B1.

If the pump does not stop when the goal level is reached – stop the pump manually and proceed with the process. If the spill tank water resistivity (R1) drop rate is high in comparison with time left for filling, stop the pump Pmp1, drain acid from T1 back into B1 by opening V13, and then drain waste water from the spill tank into T3 by opening V24. Pump the content of T3 into B2. Rinse T1 using UPW. Remove all the barrels and disassemble T1 for a leak check and repair.

The leak must be considered high if concentration of acid in the spill tank reaches 1:10 (?). This corresponds to the initial resistivity drop rate of about ??? Ohm-cm/min [See Fermilab-TM-2233].

4. Drain water from the etching tank T5 by opening V05 and V08. V05 and V08 close automatically after L5 reads zero level. Pmp5 stops automatically followed by opening V35 and V37 (automatically) to drain water remaining in the piping. Remove water from T4 by automatically opening activating Pmp4.

If V05 or V08 does not open, stop the process, drain acid back into B1 by opening V16 and V13, remove B1 from the room and made needed repair or replacement. If there is no pressure in the pneumatic line, use a spare source of compressed air to activate the valves.

5. If T3 and T4 are both empty and T2 is filled with water, open V02 and V04 to fill T5 with acid. V02 close automatically after L1 reads zero level. Start the system timer.

If V02 or V04 does not open, stop the process, drain acid back into B1 by opening V16 and V13, drain acid from the pipe by opening V07, remove B1 from the room and made needed repair or replacement. If there is no pressure in the pneumatic line, use a spare source of compressed air to activate the valves.

Close V23, open V21 and V22. Pmp5 starts automatically (accompanied by closing V35 and V37) to circulate acid through the heat exchanger HE. Monitor R6, R7, L51, L52, TG5, TG6, TG7, TG8, FM5, FM6, and FM8.

Changes in readings of R6 and R7 are indications of acid leak in the heat exchanger of in the jacket. If the leak rate in the HE is relatively high, stop Pmp 5, empty the T5 into T3 and then follow the normal rinsing procedure.

The leak must be considered high if concentration of acid in the spill tank reaches 1:10 (?). This corresponds to the initial resistivity drop rate of about ??? Ohm-cm/min [See Fermilab-TM-2233].

If the process temperature reading (TG5) becomes too high, try to increase cooling power by lowering the water chiller output temperature or increasing flow by adjusting compressed air pressure. Continue with the process unless readings of TM5 become more than 20 C. If it is, stop the process, dump acid into T3, fill the tank T5 with UPW, and start rinsing.

- 6 Fill T1 with UPW from T2 by opening V11. V11 closes automatically after the preset water level is reached.
- 7 After the process is over (timer), empty the process tank by opening V07. Pmp5 stops automatically followed by opening V35 and V37 for draining acid remaining in the piping. Stop Pmp6. Stop water chiller.

If V07 does not work, use V06, V04 and V08 to dump acid.

Close V07. Close V21 and V22 and block them from opening. Open V23.

8. Fill the etching tank with water from the tank T1 by opening V02. V02 closes automatically when T1 is empty. Pump V35 and V37 close and Pmp5 starts automatically to rinse T5.
9. When a preset value of acid is reached in T3, V29 opens and Pmp3 activates (automatically) to remove acid from T3 into B2. After the T3 is empty, Pmp3 shuts down and valve V29 closes.

If something goes wrong at this stage, keep acid in T3 and use T4 for all the rinsing steps that follow the etching procedure.

10. Fill T1 with UPW by opening V11.
11. In the end of the cycle, there is water in the process tank T5, which is circulated by Pmp5. There is no acid in the tank T3, it is removed into the barrel B2. Tank T1 is filled with slightly acidic water. Tank T2 is filled with UPW.

First Rinse

In the end of the etching cycle, there is water in the process tank T5, which is circulated by Pmp3. There is no acid in the tank T3 - it has been removed into the barrel B2. Tank T1 is filled with slightly acidic water.

The goal of the first rinse stage is making a coarse rinse following by removal of the acidic water for neutralization before releasing it into the drainage system.

1. Stop Pmp5. By opening V04, V07, V35, and V37, dump rinsing water from T5 into T3. Pmp5 stops automatically followed by opening V35 and V37. Close V04. Open V03 and V05 and fill the process tank T5 with UPW from T2. V35 and V37 close and Pmp5 starts automatically. Open V12 to fill tank T2 with UPW (V12 closes automatically after L2 shows the T2 is full). Open V27 and V30, close V28 and V29 (V25 and V29 must be blocked from opening). Start Pmp3 to rinse the piping system from remains of acid.

If V04 or V07 does not open, use V05+V08 or V04+V06+V08 circuits to dump water from the process tank (manual mode). Then proceed with rinsing.

2. Open V28, close V27, and pump out acidic water into neutralization system T9. Stop Pmp3 (automatically after level in T3 reaches zero (readings from L3). Close V28.
3. End of the First Rinsing cycle. In the end of the cycle T1 and T2 are filled with water. Nevertheless, tank T1 can have residues of acid on the upper part of its walls that will gradually dry out. Tanks T3 and T4 are empty, tank T5 is filled with water that is circulated by Pmp5.

Final Rinse

In the end of the first rinsing cycle, tank T5 is filled with water, which is circulated by Pmp5. Tanks T1 and T2 are filled with UPW.

The goal of the final rinsing cycle is to reach the stage when the process tank could be safely removed from the process room for the close loop rinsing.

1. . Open V05 and V08 to dump water from the tank T5 into T4. Pmp5 stops automatically followed by opening V35 and V37. Close V08 after reading of the level meter L5 reaches zero. Open V03 to fill the tank T5 with UPW. Pmp5 starts automatically following opening V35 and V37. Close V03 when reading from L2 shows zero.
2. Start Pmp4 to pump water out of T4 into the neutralization system. Pmp 4 shuts down automatically when L4 shows “empty”.
3. Open V12 to fill T2 with UPW. Close V12 after reading from L2 shows “full”.
4. Repeating “1”, “2” and “3” to reach neutral reaction of rinsing water ($\text{pH} > 4$). To allow for safe removal of the cavity out of the process tank.
5. In the end of the rinsing cycle tanks T1 and T2 are filled with UPW. Tanks T3 and T4 are empty. Tank T5 is filled with water.

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03/11/04

